34p.

PREPARED FOR NATIONAL AERONAUTICS AND SPACE ADMINISTRATION UNDER CONTRACT NASw-670

N64-20688

CAT. 25 CODE-1

NASA CR-56101

OCTOBER 10, 1963
JANUARY 31, 1964 -REV.

THE SYNTHESIS OF BORON CARBIDE FILAMENTS

1st QUARTERLY REPORT -REV.

OTS PRICE

XEROX $ 3.60 ph.
MICROFILM $ none

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May 26, 1964

MISSILE AND SPACE DIVISION

GENERAL ELECTRIC

R64-14342

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by

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TABLE OF CONTENTS

LIST OF FIGURES

SUMMARY

20688 A

A study has been started which has as its goal the synthesis of boron carbide whiskers, their characterization in terms of chemical and physical properties, and the utilization of such whiskers in composities.

Boron carbide whiskers have been grown successfully both by evaporation from pure B_4C vapor and by a chemical method based on the flow of appropriate gases over a heated substrate. Thus far, the best whiskers were grown by the pure vapor method. The experimental conditions required to grow B_4C whiskers, along with a description of the experimental equipment used, are presented. Included in this progress report is a short history of whisker growth and their properties in general. The expected advantages which could be realized if B_4C whiskers can be grown and utilized effectively in composites are discussed.

Future work will include a detailed study of whisker growth parameters. This will be done so that the optimum growth procedure for reproducibly high strength whiskers can be established. Such whiskers are necessary for subsequent studies of their chemical and physical properties prior to their incorporation into high strength composites. Author

I. INTRODUCTION

The severe thermal and stress problems associated with space applications has limited large advances in design and efficiency of engineering structural materials. A dire need exists for high strength, light weight materials for space structures. An approach to a solution of the space materials problem, as pertains to tension structures at least, would be the use of composite materials which could exhibit properties that no single material possesses. It has been deduced[1] that significant reduction in weight (as much as 80%) could result if ultra-strong filaments could be utilized in a composite material. Experience has already been gained with alumina whiskers* embedded in silver. Such composites have been tested at 1600°F, which is 93% of the melting point of silver. These materials were found to withstand an 82,000 psi tensile stress. A further advance in high strength silver composites, as an example, could be achieved if B_4C whiskers were available. B_4C combines a high melting point and modulus of elasticity. It also has a very high potential strength-to-weight ratio, greater than any material previously considered. Pertinent data to illustrate this fact are presented in Table I. In Figure 1 is presented a strength-versus-test temperature curve determined for α - alumina whiskers by Brenner.[2] B_4C, because of similar modulus of elasticity but higher melting point, would be expected to have similar room temperature properties but its elevated temperature strength probably would not drop as rapidly. For example, B_4C whiskers should retain significant levels of strength to nearly 4500°F, whereas α - Al_2O_3 whiskers retain their strength to about 3700°F. Thus, B_4C potentially offers substantial improvement (about 40 percent) over α - Al_2O_3 as a material for strengthening composites at high temperature.

Table I lists the densities, moduli of elasticity and melting points for a number of refractory metals and compounds. As can be noted B_4C has the highest modulus/density ratio of any material listed.

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*Naval Bureau of Weapons Contract NOw 60-0465d

TABLE I

DENSITIES, MODULI OF ELASTICITY, AND MELTING POINTS FOR A NUMBER OF REFRACTORY METALS AND COMPOUNDS (AFTER HOFFMAN, Ref. 1)

Material	Chemical Symbol	Density (lb/in^3)	Modulus of Elasticity at $70^{\circ}F$ (psi $\times 10^{-6}$)	$\left[\frac{\text{Modulus}}{\text{Density}}\right]$ (in. $\times 10^{-8}$)	Melting or Decomposition Temperature ($^{\circ}F$)
Beryllium	Be	0.066	44	6.65	2340
Boron	B	0.083	50	6.03	4200
Molybdenum	Mo	0.0369	52	1.41	4800
Tungsten	W	0.697	52	0.74	6170
Tantalum	Ta	0.601	27	0.45	5450
Columbium	Cb	0.310	23	0.74	4400
Aluminum Oxide	α - Al_2O_3	0.137	52-74	3.8-5.4	3660
Beryllium Carbide	Be_2C	0.088	45	5.08	3800
Beryllium Oxide	BeO	0.103	55	5.33	4580
Boron Carbide	B_4C	0.091	65	7.15	4500
Magnesium Oxide	MgO	0.129	12	0.93	5070
Molybdenum Carbide	Mo_2C	0.320	33	1.03	4870
Silicon Carbide	SiC	0.115	70	6.10	4350
Silicon Oxide	SiO_2	0.083	11	1.32	3150
Tantalum Carbide	TaC	0.523	42	0.80	7020
Titanium Carbide	TiC	0.178	51	2.85	5700
Titanium Oxide	TiO_2	0.170	14	0.82	3330
Thorium Oxide	ThO_2	0.346	21	0.61	5900
Tungsten Carbide	WC	0.567	102.5	1.81	5030
Zirconium Carbide	ZrC	0.242	49	2.02	6400
Zirconium Oxide	ZrO_2	0.193	36	1.87	4700
Zirconium Silicate	$ZrSiO_4$	0.154	24	1.56	4600

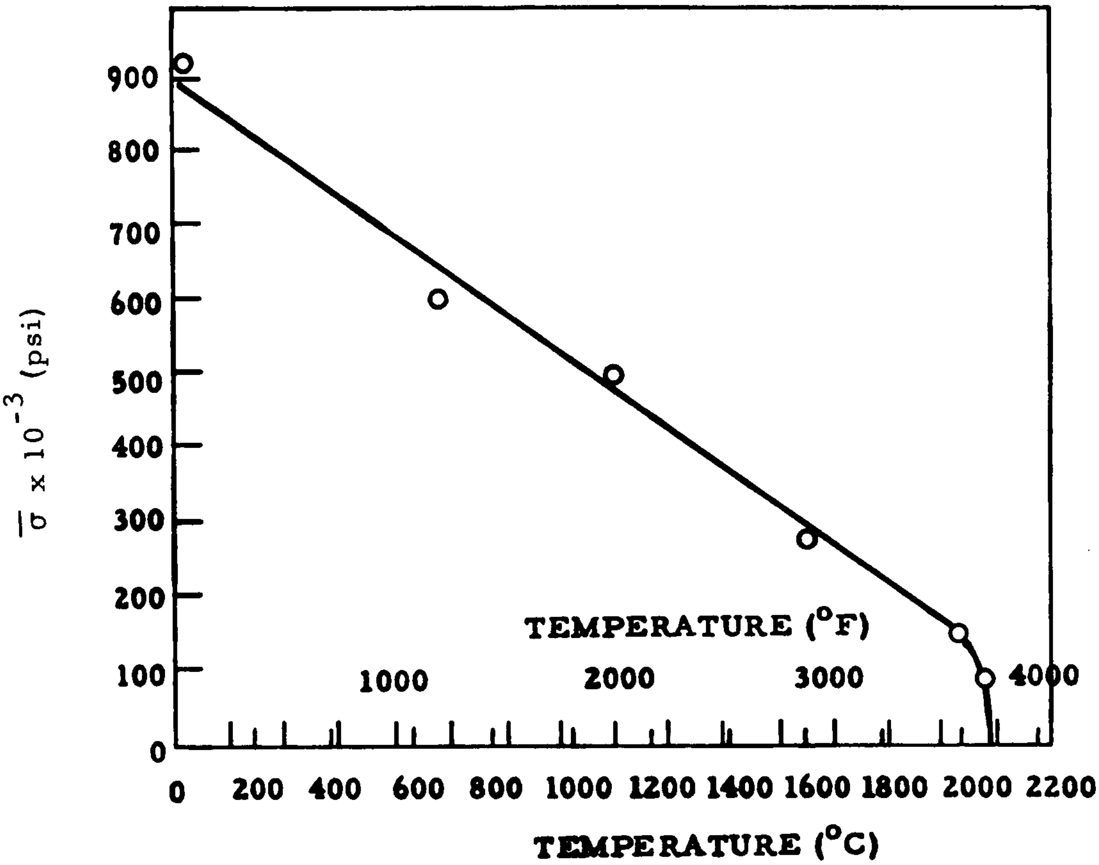

Figure 1. Average Tensile Strength of α - Al_2O_3 Whiskers

The principal emphasis during this quarter was on the synthesis of B_4C whiskers. Two growth methods were investigated. B_4C crystals were grown by both methods. A decision to pursue one specific method has been made since the whiskers produced were found to be of a size which would be more practical for use in composite materials.

II. WHISKER BACKGROUND

It has been demonstrated that the reinforcement of a metallic matrix is possible through the use of non-metallic whiskers. There are several properties of whiskers which make this possible. Foremost is their phenomenal strength which approaches the theoretical values calculated from basic considerations of the nature of bonding in a perfect crystal lattice. Values of tensile strength may be as high as 6×10^6 psi depending upon the element or compound considered.

A. THE NATURE OF WHISKERS

A scientific curiosity for many years, the filamentary crystal (whisker) is now being evaluated as a potentially useful structural material. Silver hairs which are whisker-like growths were reported by Ercker[(3)] in 1574. It is interesting to note that the discriptive nomenclature for fine, hair-like crystal forms -- whiskers -- is somewhat unscientific but most approriate. The discovery by Galt and Herring[(4)] in 1952 that tin whiskers possessed tensile strength approaching theoretical values (strength values unattained by crystalline materials prior to this time) took the whiskers from the mineralogist's curio box to the laboratory. The interest generated in whiskers has brought about much valuable knowledge and contributions to the fields of crystal growth mechanisms and of the strength of solids.

A distinction can be made between whiskers according to their origin. Whiskers can be grown from a melt; from supersaturated gas phases; from supersaturated liquid phases; from solutions; from chemical decomposition; by electrolysis or by growth from the solid. The "proper" whisker grows by the migration of atoms to the whisker base without passing through another phase (solid - phase growth). "Proper" whiskers are believed to form by surface migrations or the diffusion of atoms along a dislocation. Although many of the above modes of growth do not produce whiskers without phase change, the crystal perfection, defect structures and properties can be similar. A discussion of the many systems which produce "proper or other whiskers together with postulated growth mechanisms is not contemplated here,

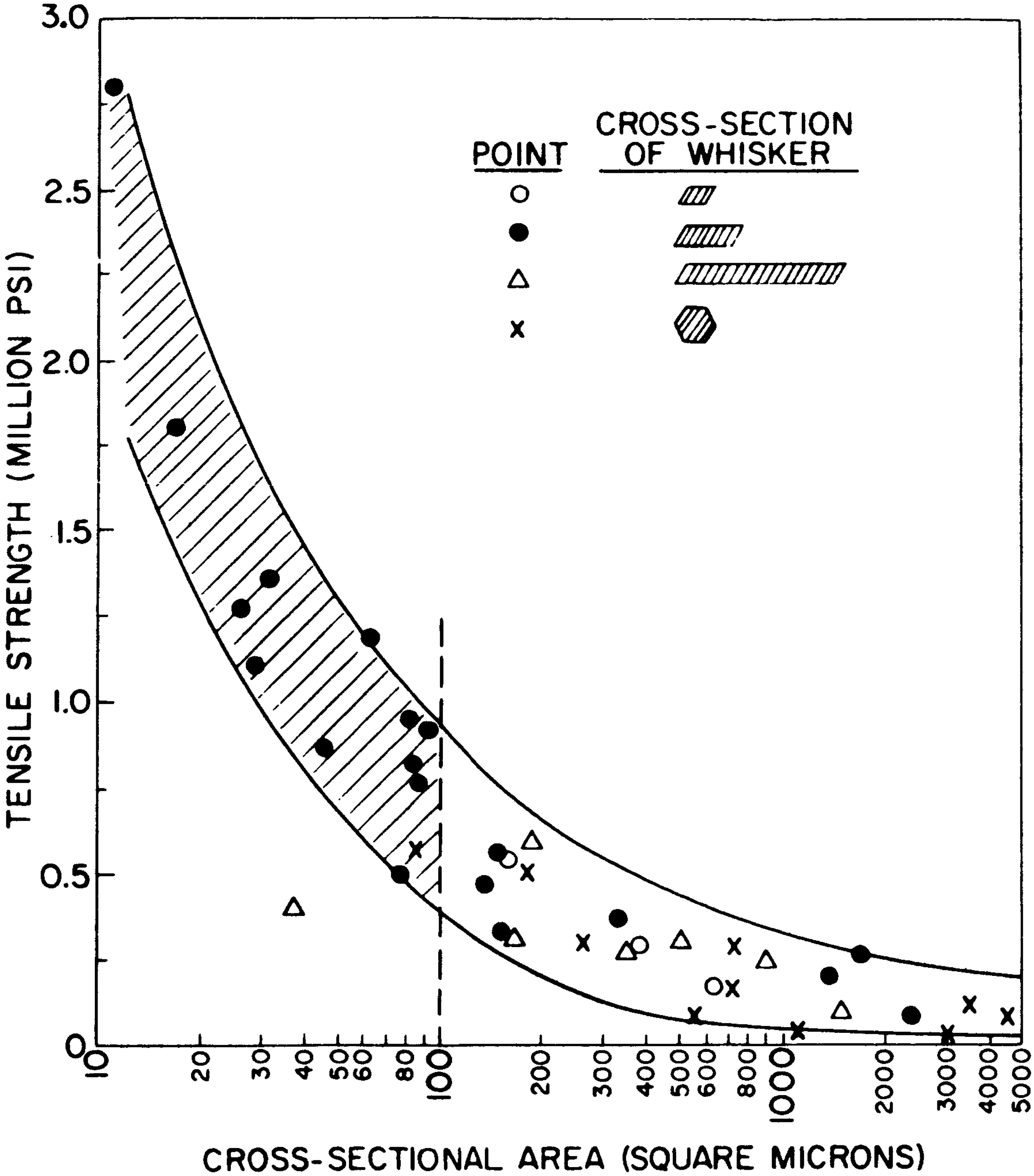

Figure 2. Relation Between Tensile Strength and Cross-Sectional Area of α - Al_2O_3 Whiskers

although many such studies and reviews are presented in contemporary scientific literature.

The morphology or form which a whisker may take can vary greatly. Shapes described as "kinked", "spiralled", "twisted", "hollow", etc have been reported. However, the high strength whiskers of most practical interest are solid and straight with a large length-to-diameter ratio. Whisker diameters can vary from submicron size to many thousands of microns and the whiskers may be many centimeters in length. Most whiskers are single crystals with their principal geometric axis parallel to a prominent crystallographic direction.

Most tensile strength determinations have been made on straight specimens. In general, as seen for alumina whiskers (Figure 2), the strength of whiskers increases as the whisker diameter decreases. The strength of whiskers has been found to be 100 to 1000 times stronger than larger bulk crystals of the same composition. One prominent theory of whisker strength is dependent on the degree of crystal perfection possible in such micro-crystals. Variations of strength between whiskers can then be rationalized by postulating slight differences in crystal and surface perfection, purity, surface films, etc.

Studies on the growth and properties of whiskers is a subject of much current interest. Much data are being accumulated but the variations of growth systems and growth conditions appear endless. A great deal remains to be learned regarding the kinetics and mechanisms of whisker growth. However, studies are being conducted to investigate process variables whereby whiskers can be grown in sufficient quantity and quality.

B. GROWTH OF WHISKERS

1. Growth Mechanism

A widely accepted mechanism of whisker growth is attributed to Sears[5]. By applying the dislocation theory of crystal growth[6], he proposed that whiskers contain a screw dislocation along the crystal axis which emerges at the tip. The whisker extends in length by the addition of atoms to the protruding terminal of the screw dislocation while maintaining smooth lateral surfaces. The Sears hypothesis contains the following criteria (a) that only one screw dislocation is present parallel to a major crystal axis and (b) during growth, the crystal is unable to nucleate new layers on its lateral surfaces due to insufficient supersaturation of the vapor.

Sears[7] has shown that the critical supersaturation criteria is valid for the growth of whiskers, from the vapors of the following materials: mercury; silver; zinc; cadmium; and calcium sulfide. Indirect evidence of the presence of screw dislocations within various whiskers has been presented by others, most notably by Webb and Forgeng[8] for alumina whiskers.

There are, however, many experimental observations which are inconsistent with the Sears hypothesis. Notable among these are (a) the need for specific surface activated impurities to produce small diameter high surface perfection whiskers[9], (b) the presence of steps on the surfaces of the whiskers as indicated by their tapers and overgrowths[9] and (c) the absence of the elastic twists in most whisker materials as would be required for the presence of axial screw dislocation[10]. The presence of steps suggests that their generation is not the rate limiting process in their growth and the absence of the elastic twist would require a much more elaborate dislocation network if dislocation growth is involved. Additional critical experiments will be required before the exact mechanism of whisker growth from the vapor can be understood.

The growth of B_4C whiskers in the present study was accomplished utilizing the technique developed by Sears for his studies of growth mechanisms. The essential features of the growth experiments are divided into two approaches; namely, (1) growth by the vaporization of bulk B_4C and (2) growth by the dynamic flow of gases containing the atomic species essential to the formation of B_4C. There are other methods which could conceivably be used to grow B_4C whiskers.

C. WHISKER USE

Refractory inorganic single crystal whisker materials have such pehnomenal strength and thermal stability at elevated temperatures that, depending on cost and availability, they will probably find many applications in rockets and missiles, nuclear reactors, supersonic jet aircraft and other types of military weapons (it is not intended to imply that military applications would necessarily be the only potential for whiskers). The main potential use for whiskers is for the reinforcement of metals, plastics and ceramics for both low and high temperature applications. Certain whisker materials, such as the refractory oxides, are usable as heat insulators; other whisker materials may qualify as special liquid and gas filters, and for use in instruments. With whisker composites, it appears reasonable that structural materials may be developed which possess at least an order of magnitude increase in tensile properties compared with the best materials available today, (for use in both ambient and elevated temperature applications). Critical applications other than those of reinforcement may some day also become important, if the problems of availability and cost are solved.

D. OBJECTIVES AND APPROACHES

The development of high strength B_4C whisker reinforced composites cannot be achieved without an adequate supply of reproducibly high strength whiskers. Therefore, a systematic study of the growth parameters for this material is actively being conducted. The specific approach, is that of studying the formation of B_4C whiskers by the two aforementioned vapor methods. A study of the deposition of whiskers from the pure B_4C vapor is progressing. By far, the best B_4C whiskers to date have been made using this technique.

Although whiskers can be produced from the pure vapor, a process based on a chemical reaction in the vapor state would be advantageous from the standpoint of the lower reaction temperatures involved, higher deposition rates (because of potentially higher operating pressures) and more precise control of the gaseous species. Therefore, the chemical method which involves the simultaneous flow across a heated substrate of various gases (containing the essential components necessary to produce the final stoichiometric B_4C) is being studied. Because this latter method has many process variables, much experimentation is required to establish optimum growth conditions. Thus far, only limited success has been achieved. Therefore, it was decided that the pure vapor deposition method for growing B_4C whiskers would be presently used as a means of accumulating a supply of B_4C whiskers. In addition, investigation of the potentially higher production chemical method will continue.

III. EXPERIMENTAL PROCEDURES

A. DESCRIPTION OF EQUIPMENT

Two high temperature resistance heated vacuum deposition furnaces are being used for this program. These furnaces are of a unique design that provide a long, very uniform (within $\pm$ 5^{o}C) hot zone at temperatures up to 2400^{o}C.

The larger of these two furnaces (Fig. 3) has a hot zone 4-1/4" in diameter by 9" long and the furnace is pumped by a 50 c.f.m. Welsh Model 1398 mechanical pump. This pump can evacuate the furnace to a pressure of less than 1 micron (10^{-3} Torr) as monitored by a Pirani gage. The graphite heating element of this furnace is operated at low voltage and high amperage by a combination of transformers controlled by a saturable core reactor. The element is insulated with graphite felt and the entire outer jacket of the furnace is water cooled. Two quartz viewing ports, one on the top and one on the side of the furnace, allow observation of the inside of the furnace and the outside of the heating element respectively. The temperature is monitored with an optical pyrometer.

The smaller of the two furnaces (Fig. 4) has a hot zone 1-3/4" in diameter by 7" long, and is pumped by a 5 c.f.m. Welsh mechanical pump. This furnace is similar to the one described, except that it is smaller. The small furnace is provided with rotometers, valving and manifolds to allow combinations of gases to be admitted to the furnace during operation. It also has a high temperature boiler pot to facilitate the introduction of less volatile compounds to the furnace.

B. MODES OF OPERATION

The two furnaces described above are being used concurrently to investigate boron carbide whisker growth. Two methods are being evaluated, the pure vapor method and the chemical method.

1. The Pure Vapor Method

In this method boron carbide is vaporized, and then recondensed in the form of whiskers. At the bottom of the large furnace, a 1" inside

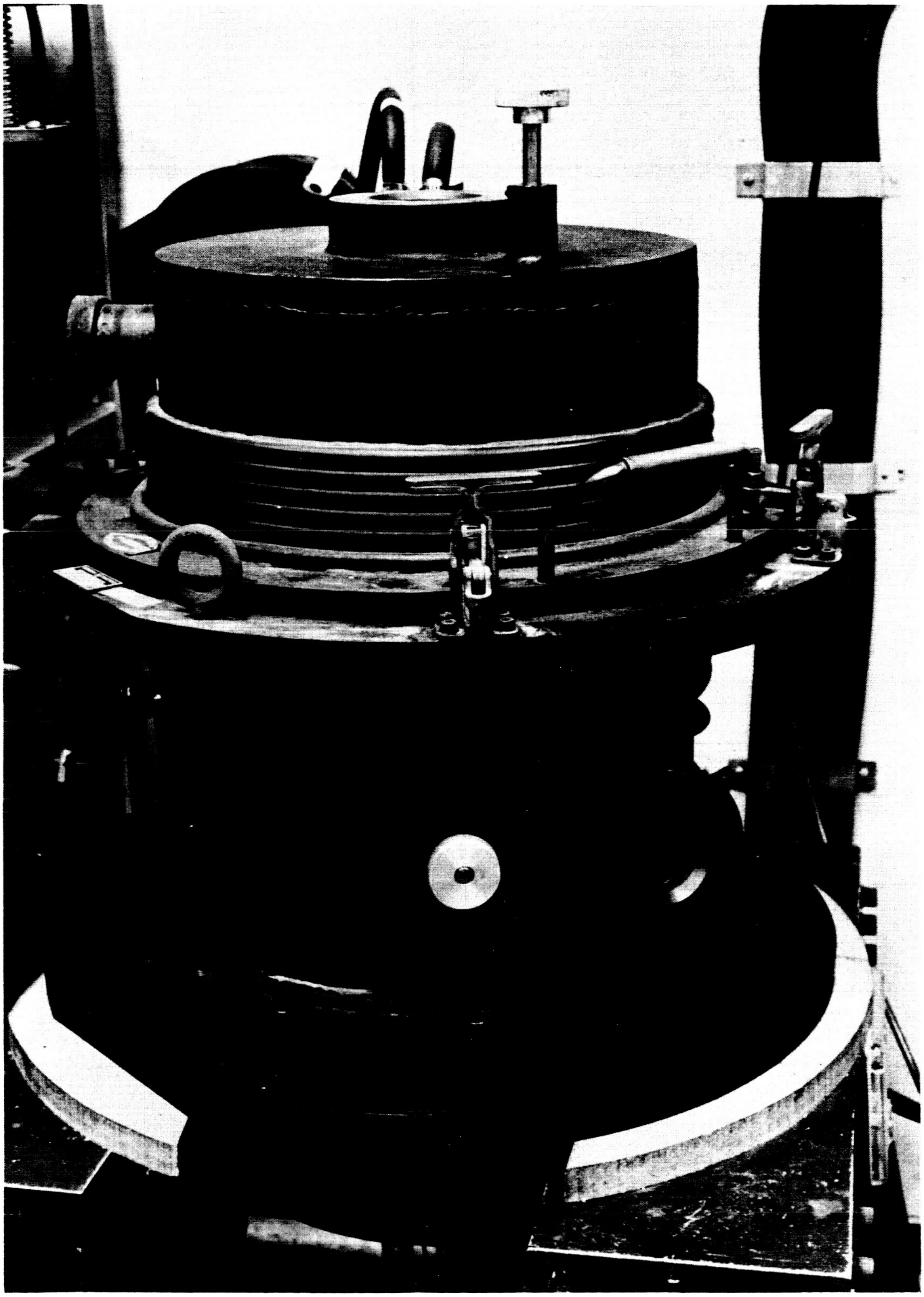

Figure 3. Photograph of Large Graphite Resistance Heated Furnace with Hot Zone 4 1/4'' Diameter by 9'' Long (About 1/4 Actual Size)

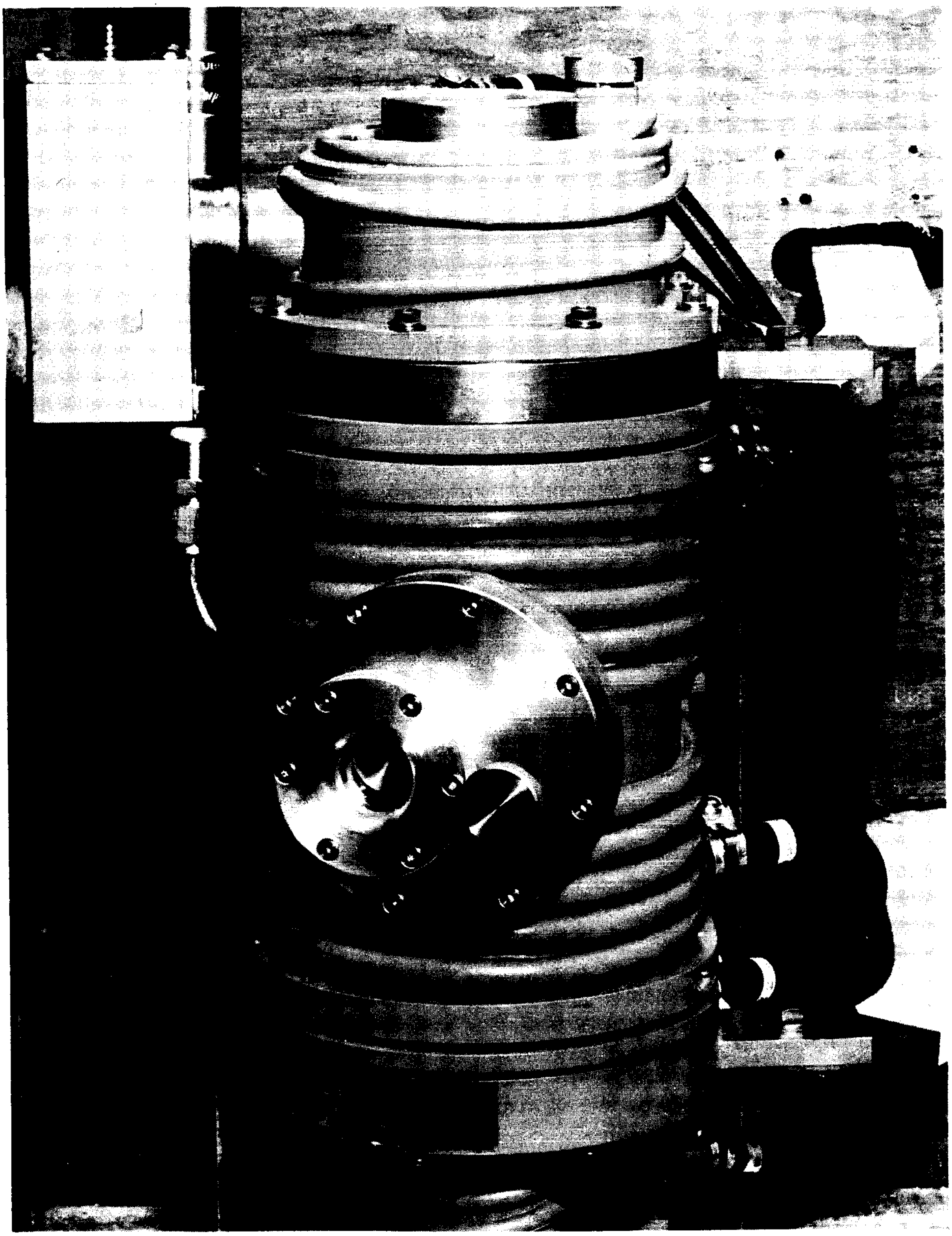

Figure 4. Photograph of a Smaller Graphite Resistance Heated Furnace with Hot Zone 1 3/4" Diameter by 7" Long (About 1/2 Actual Size)

diameter by 3/8" deep ATJ graphite cup filled with boron carbide powder (Fisher Scientific Co., -320 mesh) is placed on a pedestal 2 inches into the hot zone. An ATJ graphite deposition mandrel (Fig. 5) 3" in diameter by 9" long reduced to a 1-3/4" diameter by 5-1/4" long chimney is placed over the cup in the furnace. The chimney section extends out of the hot zone and hence provides a temperature gradient at the upper end of the furnace. Typically when the furnace is operated at 1900°C, the temperature in the bottom one inch of the chimney section is 1700°C thus providing a cooler area for the deposition of the B_4C vapor (evaporated at 1900°C). During a typical run in which B_4C whiskers are grown, the furnace pressure is approximately 75 microns.

2. Chemical Method

This method depends on the production of boron carbide in situ from a gas phase reaction such as:

$$4BCl_3 + CH_4 + 4H_2 \longrightarrow B_4C + 12HCl$$

To produce whisker growths, it is usually necessary (1) to have a suitable substrate on which the whiskers can nucleate and (2) to add a catalyst or impurity to promote the anisotropic growth.

A 1" diameter by 8" long ATJ graphite deposition mandrel is placed in the small furnace and varying flows of boron trichloride (Olin Matheson-Technical grade), methane (Olin Matheson-Commercial grade), and hydrogen (Burdett Oxygen Co. - 99.5%), are passed through the furnace at temperatures ranging from 1500 - 1900°C and at pressures varying from 12 mm down to as low as 18 microns. At the higher pressures and flow rates, anisotropic blades of born carbide were obtained.

IV. RESULTS AND DISCUSSION

The boron carbide whiskers produced by the evaporating technique were examined by X-ray and electron diffraction techniques to confirm their composition and structure. The results are summarized below. Photomicrographs of the whiskers are shown in Fig. 6. Thus far, the whiskers produced appear to be a mixture (about 10 to 1) of high surface perfection whiskers dispersed among quantities of whiskers covered with overgrowths. The whiskers are quite small. Their average size is about 0.5 mm in length and 10 microns in diameter. It is important to note that the size range in both length and diameter is fairly uniform. This will make future handling of the whiskers for use in composites relatively simple since sorting or grading problems will be minimized.

The growth density appears to be high, approximately 10^3 whiskers per square centimeter are present in the deposition zone. A deposition tube of the type shown in Fig. 5 was sectioned after deposition and is shown in Fig. 7. Included on this Figure are the operating conditions found to produce the best whiskers made to date, and the approximate temperature profile of the tube and zone in which whisker growth takes place. A typical area in the growth zone is shown in Fig. 8 at about 40X. It is to be noted that the whiskers tend to deposit on the peaks of the machining marks on the ATJ graphite and branch out radially.

A. B_4C WHISKER CRYSTAL STRUCTURE

X-ray and electron diffraction techniques were used to confirm that the whiskers produced by the methods described in this report were boron-carbide, B_4C. The results of this phase of the program are summarized as follows:

1. Specimen Preparation

   a. X-ray diffraction

Special specimen preparation techniques were developed to handle the small crystals (ca. 10μ diameter, 100μ length) for x-ray diffraction

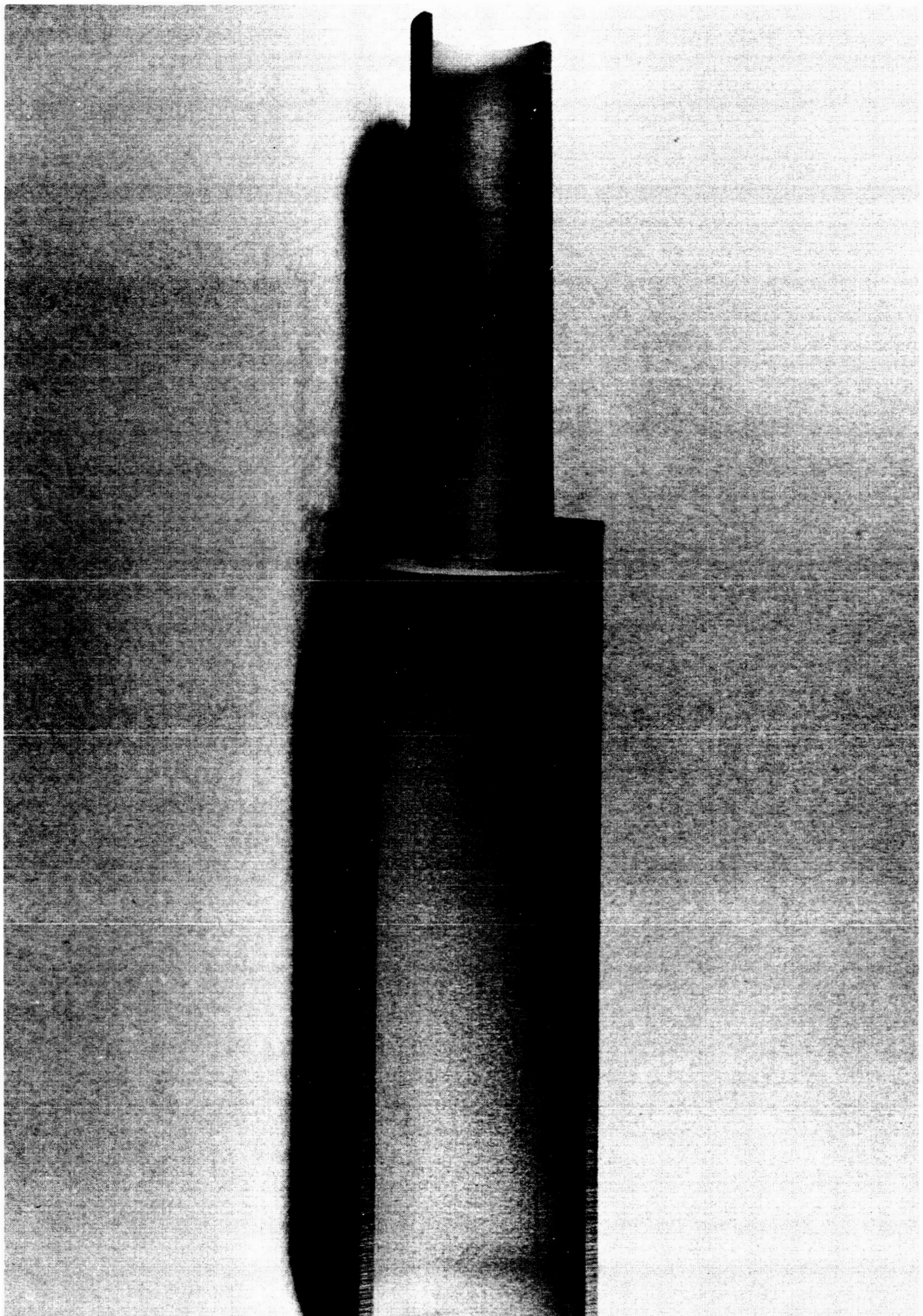

Figure 5. Photograph of Sectioned Deposition Mandrel from Large Furnace (About 1/2 Actual Size)

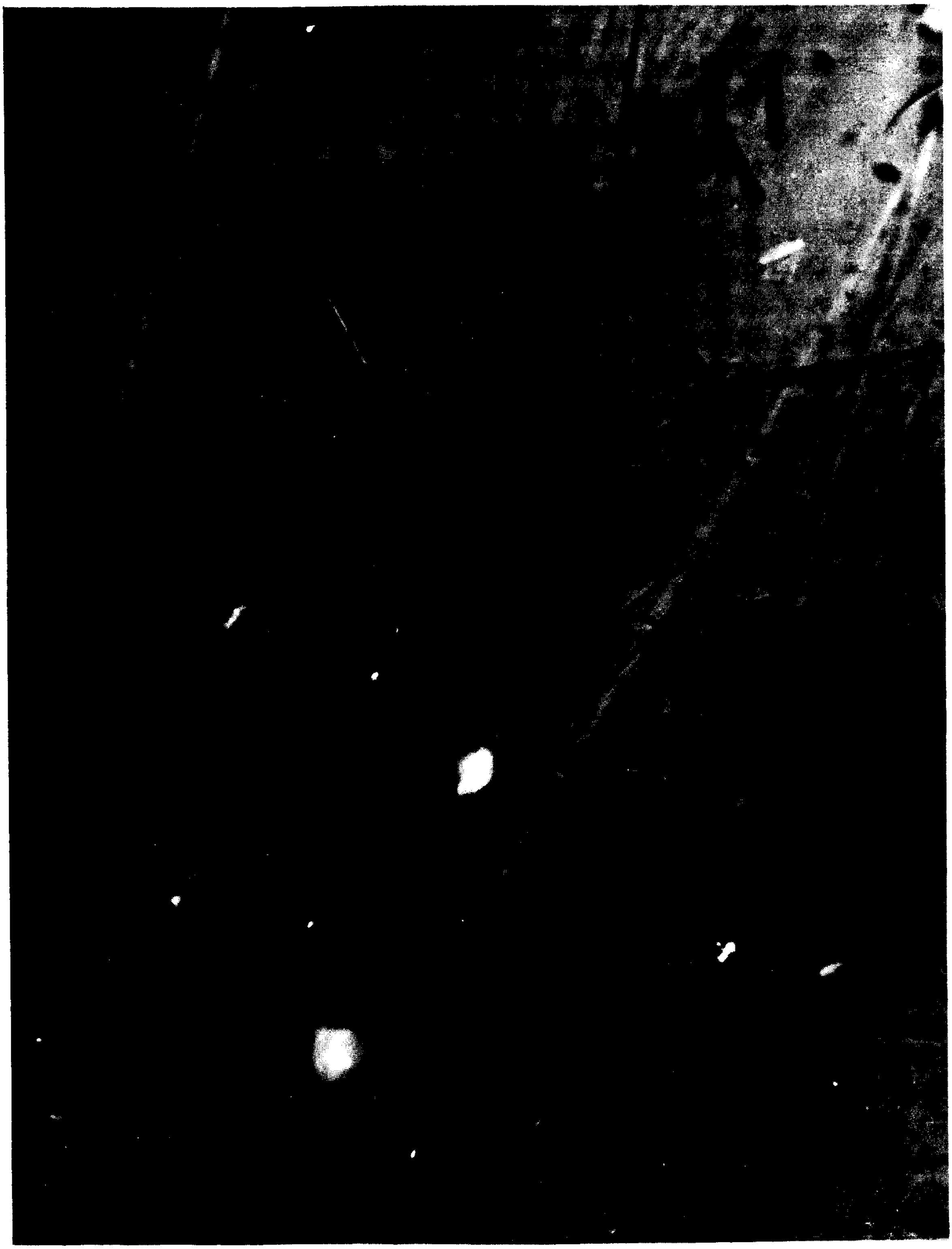

Figure 6. Photomicrographs at 500x of B_4C Whiskers

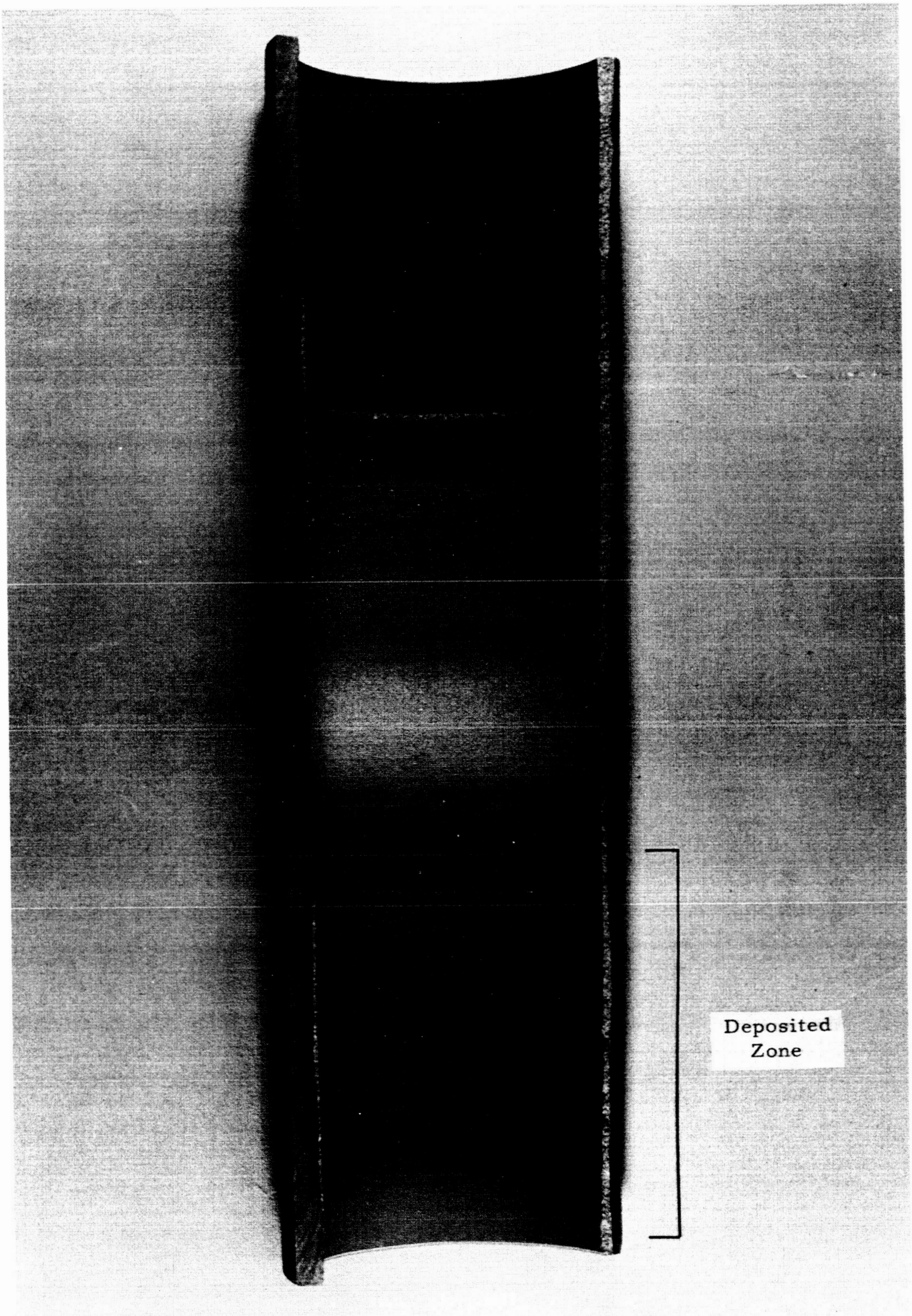

Figure 7. Section of Deposition Tube Indicating B_4C Whisker Growth Region. About 1/2 Actual Size Operating Conditions: 1700°C @ 4 hours, 75 μ pressure

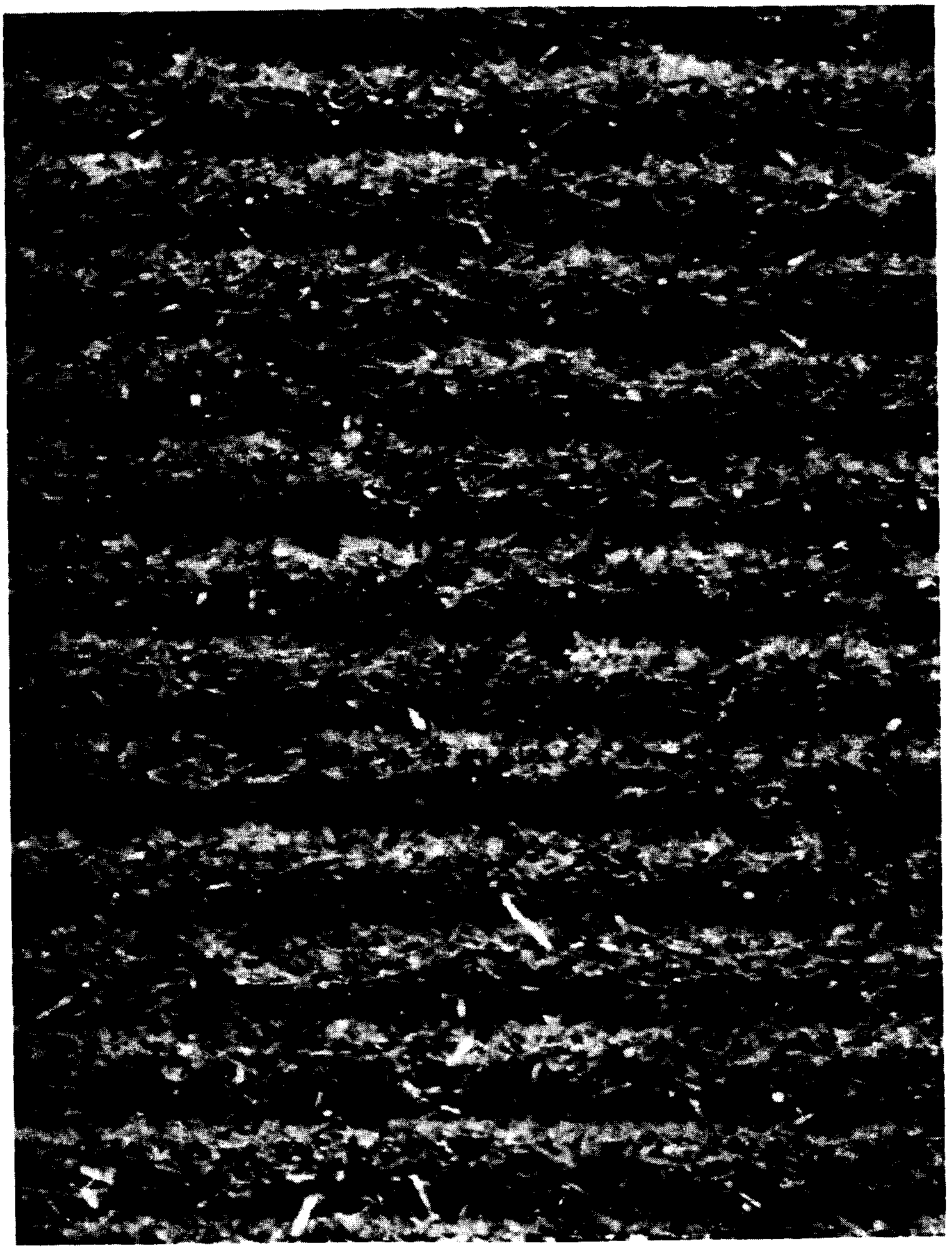

Figure 8. Deposition Zone at 40x Showing as-Deposited B_4C Whiskers

analysis. It was found that very suitable 'rat-tail' specimens could be produced by rolling out or kneading a drop of Duco cement in which many whiskers had been embedded. The whiskers were picked up in the drop of cement by manipulating the drop over the inner surface of the tube on which the whiskers had been produced. The specimens so formed were examined in a cylindrical, powder x-ray camera.

b. Electron diffraction

Specimens were very simply prepared by placing a standard copper electron microscope specimen screen, which had previously been coated with a commercial adhesive*, in contact with the surface upon which the whiskers had grown. Quantities of suitably thin (ca. 1000A thick) whiskers were collected in this manner and examined in an Hitachi HU-11 electron microscope by means of selected area diffraction (a transmission diffraction technique).

2. Results of Diffraction Studies

The results of both electron diffraction and x-ray diffraction studies indicated that the whiskers were (1) essentially boron-carbide with a stoichiometry of B_4C, (2) single crystal and (3) coated with a thin film of graphite.

Presented in Fig. 9 are (a) a transmission electron photomicrograph of a 'typical' B_4C whisker and (b) an electron diffraction pattern of the same whisker. It is to be noted that the whisker is a single crystal as evidenced by the nature of the spots in the diffraction photograph. The interplanar separations (d-spacings) as calculated from the positions of the diffraction spots on this and similar diffraction patterns qualitatively agree with those presented in the literature(11) for B_4C. In most cases, the thicker whiskers (>1000Å) are found to be coated with a thin film. Electron diffraction examination of this film, in those regions in which the film is found to be lifted off of the whisker, indicates that it is graphite.

*Adhesive was supplied by Ernest F. Fullam, Inc.

(a)

Magnification 11,000 X

(b)

Figure 9. Transmission Electron Photomicrograph of a Typical B_4C Whisker (a) and an Electron Diffraction Pattern of the same Whisker (b)

Future reports will include detailed discussions of the morphology and structure of B_4C whiskers. At such time, complete descriptions of electron microscope and electron diffraction studies will be presented.

Shown in Fig. 10 is the x-ray diffraction photograph* of a whisker specimen prepared in the manner described above. The sharp ring-like nature of the diffraction lines in this photograph indicates that the kneading operation which was employed in the preparation of the specimen has served to fracture the whiskers and has randomly dispersed the fragments within the specimen. In Table II are presented the x-ray diffraction data as obtained from the photograph in Fig. 10. The experimental data is compared in this table with the published data[11] on B_4C.

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*The photograph was prepared using nickel filtered copper radiation (λ = 1.5405A) in a 57.3 mm diameter powder camera.

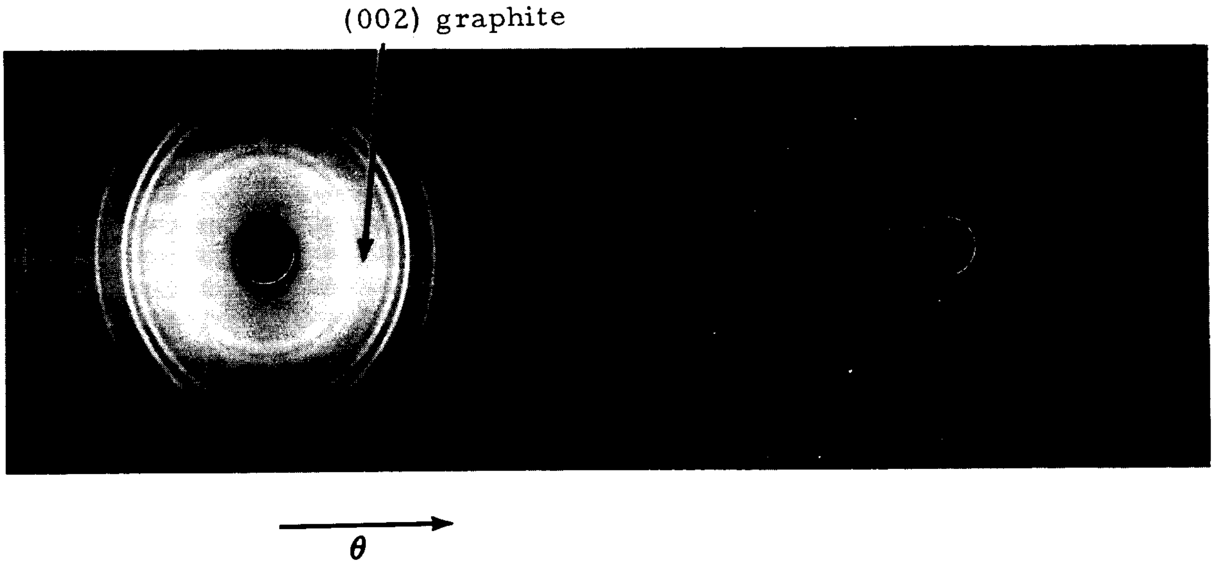

Figure 10. X-ray Diffraction Photograph From B_4C Whisker Specimens. (Cu Radiation, 57.3 mm ϕ Camera)

TABLE II
X-RAY DIFFRACTION DATA FROM BORON CARBIDE WHISKERS COMPARED WITH STANDARD DATA FOR B_4C

From Diffraction Photograph from Figure 10		From ASTM Card for B_4C (Card 6-0555)		
I	d(Å)	I	d(Å)	(hkl)
m	4.47	30	4.49	101
ms	4.05	40	4.02	003
s	3.78	70	3.79	012
ms	3.36	--	---	---
vvw	3.20	--	---	---
w	2.80	30	2.81	110
vvw	2.61	--	---	---
vs	2.57	80	2.57	104
vvs	2.38	100	2.38	021
vvw	2.31	10	2.30	113
s	2.08	10	2.02	006
vvw	1.81	10	1.82	211
mw	1.71	30	1.714	205
--	---	10	1.637	116
--	---	10	1.628	107
mw	1.50	20	1.505	303
m	1.46	30	1.463	125
m	1.44	30	1.446	018
ms	1.40	30	1.407	027
vw	1.36	20	1.345	009
w	1.33	20	1.342	131
w	1.31	20	1.326	223
vw	1.28	10	1.286	208

TABLE II (continued)

From Diffraction Photograph from Figure 10		From ASTM Card For B_4C (card 6-0555)		
I	d(Å)	I	d(Å)	(hkl)
w	1.26	20	1.261	306
w	1.19	10	1.191	042

I = relative intensity; w = weak, m = medium, s = strong, v = very

Note: B_4C is hexagonal, a_o = 5.61A, c_o = 12.07A

The excellent agreement between the experimental x-ray data and the published or reference data for B_4C indicates that the whiskers are indeed B_4C. The additional, medium strong, line (see arrow Fig. 10) with d = 3.36A can possibly be indexed as the (002) reflection for graphite (^{d}002 graphite = 3.37A). The fact that graphite appears to be present is in agreement with the before mentioned electron diffraction observations.

V. CONCLUSIONS

Initial results have been favorable and the following conclusions can be made:

1. Boron carbide, B_4C, whiskers have successfully been produced. This is the first synthesis of this material to be reported. It remains to carefully investigate the process and to adjust the various growth parameters so that reproducibly high strength whiskers of more reasonable lengths are produced.

2. Of the two growth methods studied thus far, the pure vapor technique is the more reproducible method.

VI. FUTURE WORK

The operating parameters found necessary to produce B_4C whiskers (which include temperature, time, pressure and impurity additions) by the deposition of pure vapor will be studied in detail. Goals in this work will include the growth of longer whiskers suitable for testing and their subsequent evaluation as to strength properties.

The advantages of the chemical system of whisker growth are such that more runs will be made to determine if the process can be made to work.

ACKNOWLEDGMENTS

The authors are indebted to Messrs. W. Laskow and C. Miglionico for the photographs, T. Harris for the x-ray work, Dr. P. D. Gorsuch who reviewed this report and Dr. W. H. Sutton and J. Chorne for many stimulating technical discussions.

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11. ASTM x-ray diffraction data card 6-0555.

12. ASTM x-ray diffraction data card 12-212.

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